SEQUENTIAL IMPLANTATIONS OF DEUTERIUM AND HELIUM IONS INTO TUNGSTEN-COATED COMPOSITE STRUCTURES

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The trapping and thermal desorption of deuterium and helium implanted into tungsten-coated composite structures were studied. The amount of accumulated deuterium and helium, and the shape of thermal desorption spectra were shown to be depended on the way of irradiation by the D^+ and He^+ ions: individually or sequentially. The possible mechanisms of these processes are proposed.

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INTRODUCTION

Tungsten coating of multilayer functional structures is a promising material for devices contacting with the plasma in fusion installations. The possibility of its use in such devices is largely depend on its radiation resistance to the accumulation in it hydrogen isotopes and helium, forming in the result of the interaction with plasma streams. As very actual, this problem is intensively studied in recent years [1-6]. In [1-3] a comprehensive analysis of formation of matrix radiation defects, as well as the capture, retention and thermal desorption of deuterium and helium, ion-implanted in tungsten coatings of multilayer composite systems separately, was done. It is of interest to carry out these studies at sequential implantation of He^+ and D^+ ions. Only a few literature data showed a mutual influence on each other sequentially implanted ions of hydrogen isotopes and helium in tungsten during irradiation and after heating [4-6]. In this paper we studied the influence of preimplantation of He^+ ions (or D^+) on the accumulation of deuterium (or helium) in tungsten coatings of multilayer composite structures and their thermal desorption in a vacuum. The types of radiation damage, formed during single and sequential irradiations by D^+ and He^+ ions, were cleared, their influence on the structural features and radiation resistance of tungsten coatings was shown.

1. EXPERIMENTS

Tungsten coatings of about 1 micron in thickness deposited by magnetron sputtering of W target in Ar atmosphere at the pressure of 1.0 Pa were studied. Deposition was carried out at a rate of 0.6 nm s^{-1} on a stainless steel substrate (SSt) 0.5 mm thick at a temperature T = 600 K with pre-deposited Ti layer of thickness less than 10 nm (SSt + W). Tungsten coatings have a polycrystalline structure with an average grain size of about 60 nm and a low degree of texture with texture axis [110]. The samples were irradiated at the room temperature sequentially by 10 keV D^+ (20 keV D_2^+) and 20 keV He^+ ion beams at a current density ~ 5 μA cm^{-2} up to doses Φ in the range $(0.2…5.0)\times10^{17}$ cm^{-2} in the sequence: He^+, D^+ or D^+, He^+. Mean projective and full ranges of D^+ (10 keV) and He^+ (20 keV) ions in the tungsten coatings from the calculations [7] were about 60 and 160 nm respectively; they were comparable for compared ions and significantly less than the coatings thickness. According to [7], profiles of W lattice radiation damages, generated by D^+ and He^+ ions, were identical and situated in the area of implanted ions. In experiments with implantation of only one type of ions their energy and dose were the same as at combined sequential implantations.

In the studies we used the methods of thermal desorption spectrometry (TDS) (on PTI-7A gas mass spectrometer, with calibrated helium leak valve GELIT-1) and electron microscopy. We got the spectra of thermal desorption of helium and deuterium and, using the data of the cross section of ionization of helium and deuterium particles [8] in the source of the mass spectrometer, determined the concentrations C and capture coefficient of implanted gases $\eta = C/\Phi$. A more detailed description of the experimental procedure was given in [9].

The desorption spectra of helium atoms and deuterium molecules were got, heating the irradiated samples with a constant rate $\alpha = 0.8$ K·s^{-1} in the temperature range 290…1800 K. The temperature measurements error was ± 5 K. The spectra represented dependencies of the number S of implanted gas particles, released at the heating temperature T, on this temperature. The sensitivity of the used method of determining the number of helium and deuterium particles was not worse than 2×10^{12} cm^{-2}. When heated the non-irradiated samples the partial pressures of helium and deuterium were less than 1×10^{-7} Pa. Initial background of particles with $m = 4$ was not greater than $S = 0.001\times10^{16}$ cm^{-2} at $T < 1200$ K and $S < 0.005\times10^{16}$ $см^{-2}$ in the temperature range ΔT = 1600…1800 K, where the evaporation of stainless steel substrate components and a slight increase in the residual vacuum in the chamber were observed. In the thermal desorption spectra of helium and deuterium implanted separately, at $T < 1200$ K the value S for the helium did not exceed the background S, and the S value for the deuterium at $T \geq 1200$ K also did not exceed the background value. The latter fact allowed to suggest that the thermal desorption spectra of helium and deuterium ions which were implanted in sequential

combinations, at $T < 1200$ K showed the D_2 release from the W coating in vacuum, and He release when $T \geq 1200$ K.

Studies of changes of microstructures of the W coating near surface layers with implanted helium and deuterium were done on thinned samples using a transmission electron microscope TEM-100 L. The morphology of the surface of the irradiated samples was monitored with a scanning electron microscope SEM -100 U.

2. RESULTS AND DISCUSSION

When heated the composite structures with tungsten coatings single or sequentially irradiated by D^+ and He^+ ions, thermal release of implanted gases into the vacuum was observed. In Fig.1 the spectra of deuterium thermal desorption from tungsten coated composite structure (SSt + W (μm)), irradiated only by D^+ ions up to different doses are shown. The desorption of helium from the tungsten coating of the same composite structure, irradiated only by He^+ ions to different doses, are shown in Fig. 2. In Fig. 3 there are shown the spectra of thermal desorption of deuterium and helium from the tungsten coated composite structure (SSt + W (μm)), irradiated sequentially D^+ and He^+ ions under the schemes: $(He^+ – D^+)$ – curve 1 and $(D^+ – He^+)$ – curve 2. For other doses of the sequential irradiations by the named ions spectra of thermal desorption of deuterium and helium were similar.

As seen from Fig. 1, a significant deuterium release from the coatings started at temperatures $T \geq 350$ K, and ended at $T \approx 1000$ K (curves 1-4). At $\Phi_{D^+} \geq 5.0 \times 10^{17}$ cm^{-2} small D_2 release took place up to $T \approx 1200$ K (curve 5). There was one peak of deuterium thermal desorption with the maximum temperature T_m near 640 K.

As seen from Fig. 2 (curves 1-4), the temperature range of helium release $\Delta T \approx (900…1800)$ K did not agree with ΔT of deuterium. Helium released mainly at $T_m \approx 1500$ K. At $\Phi_{He^+} \geq 4.7 \times 10^{17}$ cm^{-2} (curves 5, 6) in the spectra of helium thermal desorption there appeared the low-temperature region of this gas release: $\Delta T \approx (350…900)$ K, which superposed with the area of D_2 release (see Fig. 1). From a comparison of Figs. 1-3 it could be concluded that, if the irradiation doses did not exceed 5.0×10^{17} cm^{-2}, the spectra of thermal desorption of deuterium and helium ions which had been implanted in different sequences represented a superposition of spectra of thermal desorption D_2 and He, implanted with the same doses separately. The temperature ranges of deuterium and helium release remained the same; maxima of peaks of thermal desorption had the same T_m both for single and sequentially implantations of D^+ and He^+ ions.

Fig. 4 shows the dependences of the concentration C_{He} (1) and capture coefficient η_{He} (2) of helium in the tungsten coating on the dose Φ_{He^+} of He^+ ions irradiation in different schemes (only He^+ – ●, ▲; sequentially: $(He^+ – D^+)$ – ○, Δ, or $(D^+ – He^+)$ – ×, ⁎). Fig. 5 shows the dependences of the C_D (1) and η_D (2) of deuterium on the dose Φ_{D^+} of D^+ ions irradiation in various schemes (only D^+ – ●, ▲; sequentially: $(He^+ – D^+)$ – ○, Δ, or $(D^+ – He^+)$ – ×, ⁎). As can be seen from Figs. 4 and 5, for different schemes of D^+, He^+ ions irradiation the values C_{He} and η_{He} for implanted helium much more than similar values of C_D and η_D for implanted deuterium.

The dependences in Fig. 6 show the influence of preimplantation of one type of gas on accumulation of another gas in sequential irradiation of tungsten coatings

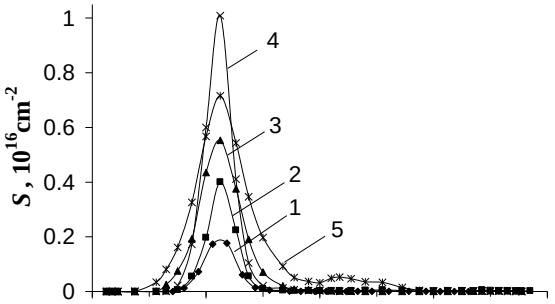

Fig. 1. Spectra of deuterium thermodesorption from the tungsten coatings of the composite structure (SSt+ W(μm)) that irradiated by D^+ ions (10 keV, Φ_D+, 10^{17}cm^{-2}: 1 – 1.0; 2 – 2.0; 3 – 3.0; 4 – 4.0; 5 – 5.0); α = 0.8 K·s^{-1}

by D^+, He^+ ions.

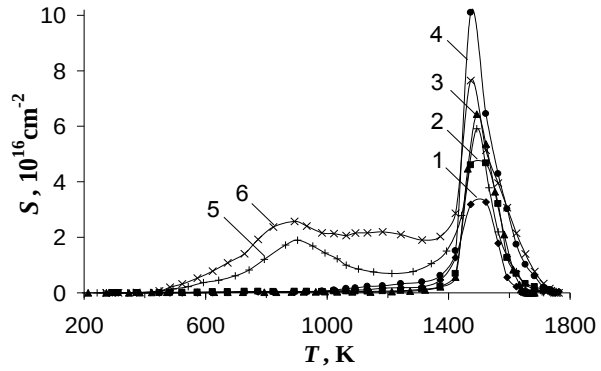

Fig. 2. Spectra of helium thermodesorption from the tungsten coatings of the composite structure (SSt+ W(μm)) that irradiated by He^+ ions (20 keV, Φ_{He^+}, 10^{17}cm^{-2}: 1 – 1.0; 2 – 2.0; 3 – 3.1; 4 – 4.0; 5 – 4.7; 6 – 8.3); α = 0.8 K·s^{-1}

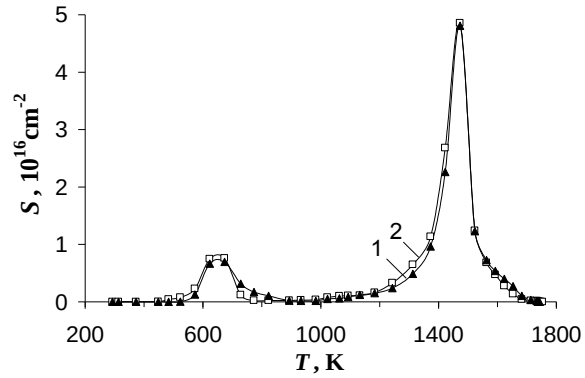

Fig. 3. Spectra of deuterium and helium thermodesorption from the tungsten coating of the composite structure (SSt.+ W(μm)) that were sequentially irradiated by:$(He^+ – D^+)$ – curve 1; $(D^+ – He^+)$ – curve 2; D^+ (10 keV, 2.0×10^{17}cm^{-2}); He^+ (20 keV, 2.0×10^{17}cm^{-2}); α = 0.8 K·s^{-1}

As followed from the results, pre-implantation of helium to different doses did not change the values of C_D and η_D for deuterium (see the dependences $C_D = f(\Phi_{He^+})$ (curve 1) and $\eta_D = f(\Phi_{He^+})$ (curve 2) in Fig. 6). Deuterium pre-implantation to different doses also did not change the values of C_{He} and η_{He} for helium (see the dependences $C_{He} = f(\Phi_{D^+})$ (curve 1') and $\eta_{He} = f(\Phi_{D^+})$ (curve 2') in Fig. 6.

In the works of other authors [4-6] for W foil there was shown the influence of pre-implantation of He^+ ions with the scheme ($He^+ - D^+$) on the capture of deuterium. Pre-irradiation of He^+ ions (8…10 keV) at $0.3 \le \Phi_{He^+} \le 1.0\times10^{17}$ cm^{-2}, did not change the retention of D^+ ions, implanted at $\Phi_{D^+} = (1–2)\times10^{17}$ cm^{-2} [4] and increased it by 3 times, when the dose of He^+ ions reached $\Phi_{He^+} = 2.0\times10^{17}$ cm^{-2} [5]. The authors connected these facts with the capture of D particles in radiation defects of matrix in the first case [4] and the influence of a field of strong tension around individual helium bubbles in the second [5]. According to [6], with a further increase of the dose of He^+ ions (3 keV) up to $\Phi_{He^+} = 1.0\times10^{19}$ cm^{-2}, the capture of deuterium decreased in the result of the influence of the formed network of helium bubbles on the accumulation of deuterium. Authors [6] noted a decrease of deuterium capture in sequential implantation (D^+–He^+) ($\Phi_{D^+} = 1\times10^{17}$cm^2), $\Phi_{He^+} = 1.0\times10^{17}$cm^{-2}) in the result of radiation enhanced diffusion of weakly retained deuterium atoms to the surface of the sample and released in the vacuum at the further irradiation by He^+ ions.

TEM studies of the authors of this paper [2, 3] of tungsten coatings single irradiated at room temperature by He^+ ($\Phi_{He^+} \le 2.0\times10^{17}$ cm^{-2}) or D^+ ($\Phi_{D^+} \le 6\times10^{18}$ cm^{-2}) ions showed that deuterium and helium bubbles did not formed in the grains of the crystal structure. Helium bubbles were visible at $\Phi_{He^+} \ge 7\times10^{17}$ cm^{-2}, they have the average diameter and density 2.5 nm and 5×10^{12} cm^{-2} respectively at $\Phi_{He^+} = 7\times10^{17}$ cm^{-2}. It was also observed the formation of interstitial dislocation loops and dislocation networks with an average diameter > 5 nm and a density greater than 3.2×10^{12} cm^{-2}. On the obtained data the following types of radiation defects, formed in the volumes of tungsten coatings under sequential irradiation by He^+ and D^+ ions, were proposed: vacancy-type defects, interstitial dislocation loops, gas-vacancy complexes He_mV_n, D_mV_n.

CONCLUSIONS

The radiation resistance of tungsten coatings composite structure (SSt + W (μm)) to the sequential irradiation by D^+ and He^+ ions of medium energies up to doses $\Phi_{D^+} \le 4{,}0\times10^{17}$ cm^{-2} and $\Phi_{He^+} \le 4{,}7\times10^{17}$ cm^{-2} at the room temperature was studied. The spectra of the thermal desorption of deuterium and helium from the sample in vacuum was analyzed; capture coefficients for these gases in the coatings were determined. At single and sequential implantation of D^+ and He^+ ions in tungsten coatings such regularities were established: deuterium was accumulated in tungsten coatings at lower concentrations compared with helium, and its capture coefficient was about one order of magnitude lower.

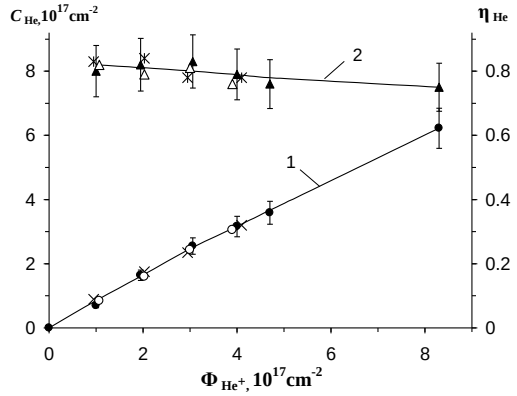

Fig. 4. Dependences of the concentration C_{He} (1) and capture coefficient η_{He} (2) of helium particles in the tungsten coating of the composite structure on Φ_{He^+} fluency for different ways of irradiation: ●, ▲–He^+ ions; ×, ж– (D^+– He^+) ions; ○, Δ – (He^+– D^+) ions; D^+ (10 keV, 2.0×10^{17}cm^{-2}); He^+ (20 keV)

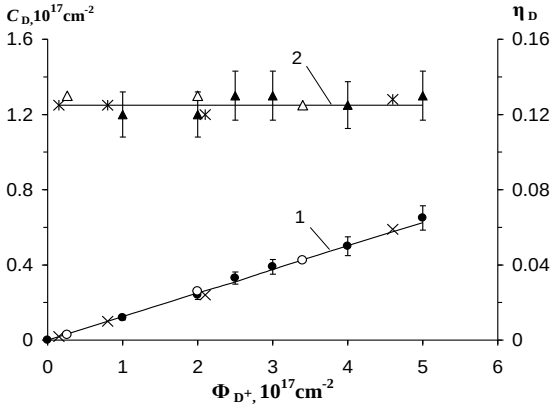

Fig. 5. Dependences of the concentration C_D (1) and capture coefficient η_D (2) of deuterium particles in the tungsten coating of the composite structure on the Φ_{D^+} fluency for different ways of irradiation: ●, ▲ – D^+ ions; ×, ж – (D^+ – He^+) ions; ○, Δ – (He^+ – D^+) ions; He^+(20 keV, 2.0×10^{17} cm^{-2}); D^+ (10 keV)

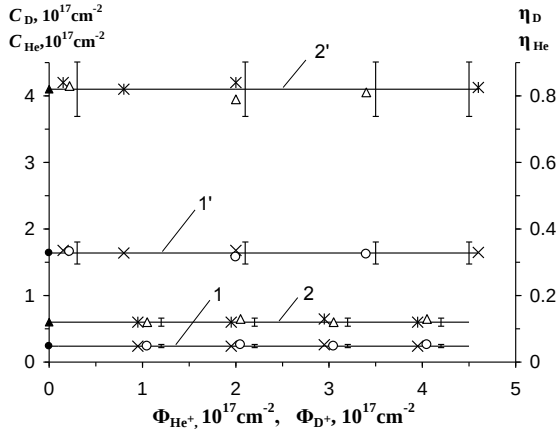

Fig. 6. Dependences of $C_D = f(\Phi_{He^+})$ (curve 1) and $C_{He} = f(\Phi_{D^+})$ (curve 1'), $\eta_D = f(\Phi_{He^+})$ (curve 2) and $\eta_{He} = f(\Phi_{D^+})$ (curve 2') for D_2 and He in the tungsten coating irradiated: single D^+ or He^+ ions – ●, ▲; (He^+– D^+) sequentially – ○, Δ; (D^+– He^+) sequentially – ×, ж. The curves 1, 2 for $\Phi_{D^+} = 2.0\times10^{17}$ cm^{-2} and the curves 1', 2' – for $\Phi_{He^+} = 2.0\times10^{17}$ cm^{-2}

At sequential implantations of these ions the temperature range of deuterium or helium release and the temperatures of the peaks maxima in the spectra of thermal desorption were the same as in the single implantation; accumulation of deuterium (helium) was independent of He^+ (D^+) ion pre-implantation. It was suggested that into tungsten coatings under sequential irradiations the following types of radiation damage were formed: defects of the vacancy type, interstitial dislocation loops, gas – vacancy complexes He_mV_n, D_mV_n.

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ПОСЛЕДОВАТЕЛЬНАЯ ИМПЛАНТАЦИЯ ИОНОВ ДЕЙТЕРИЯ И ГЕЛИЯ В ВОЛЬФРАМОВЫЕ ПОКРЫТИЯ КОМПОЗИЦИОННЫХ СТРУКТУР

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Изучены процессы захвата и термической десорбции дейтерия и гелия, имплантированных в композиционные структуры с вольфрамовым покрытием. Количества накопленных дейтерия и гелия и вид спектров термической десорбции показаны в зависимости от схемы облучения ионами D^+ и He^+: по отдельности или последовательно в разной очерёдности. Предложены возможные механизмы этих процессов.

ПОСЛІДОВНА ІМПЛАНТАЦІЯ ІОНІВ ДЕЙТЕРІЮ ТА ГЕЛІЮ У ВОЛЬФРАМОВІ ПОКРИТТЯ КОМПОЗИЦІЙНИХ СТРУКТУР

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Вивчено процеси захоплювання та термічної десорбції дейтерію та гелію, імплантованих у композиційні структури з вольфрамовим покриттям. Кількості накопичених дейтерію та гелію та вигляд спектрів термічної десорбції показано в залежності від схеми опромінення іонами D^+ та He^+: окремо або послідовно в різній черговості. Запропоновані можливі механізми цих процесів.